

REMARKS

Favorable reconsideration is respectfully requested.

The claims are 17-28.

Claims 17-28 have been rejected under 35 U.S.C. § 112, second paragraph as indefinite in the term "amide I set to 1".

It is stated that the specification does not provide any guidance as to this concept.

In reply, please see the Disclosure on page 16, lines 2-19 of the present specification in which this matter discussed in detail.

Accordingly, the rejected terminology is definite.

Claims 17-22 have been rejected under 35 U.S.C. § 102(b) as anticipated by or, in the alternative, under 35 U.S.C. § 103(a) as obvious over Hojo et al. (U.S. 5,824,113).

Further, claims 23-28 have been rejected under 35 U.S.C. § 103(a) as being unpatentable over Hojo et al. as applied to claims 17-22 above, and further in view of Thorsen (U.S. 4,189,303).

These rejections are respectfully traversed.

1. INTRODUCTION

The Final Rejection is different from First Action only in that the Final Rejection states that "In examples 3 and 6, Hojo et al. illustrate the use of a NaCO₃ aqueous solution and the use of NaOH for the purpose of removing the water repellent keratin scales. The examiner asserts that the teachings of Hojo et al. illustrate removal of the keratin layer and the OPTIONAL removal of the under-keratin layer the property of water repellency is not wanted in the fabric. Please see examples 3 and 6 and part c) in column 3."

In reply, the present invention and Hojo's invention will be explained to enable the Examiner to fully understand the difference between the present invention and Hojo's invention. Firstly, it should be emphasized that the product and the production method of the present invention are completely different from those of Hojo et al. When this difference is understood, it will be seen that the present invention is completely unobvious from Hojo et al. and Thorsen.

2. CONSTITUTION OF EPIDERMIS IN WOOL

The constitution of Epidermis in wool fiber is shown in Fig. 1(A)-(C) below.

Fig. 1(A)

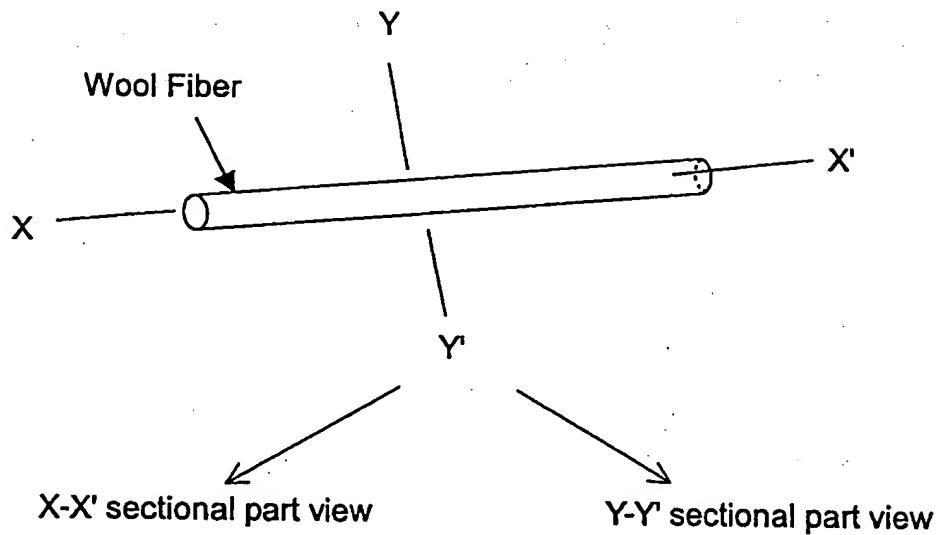


Fig. 1(B) (X-X' sectional view)

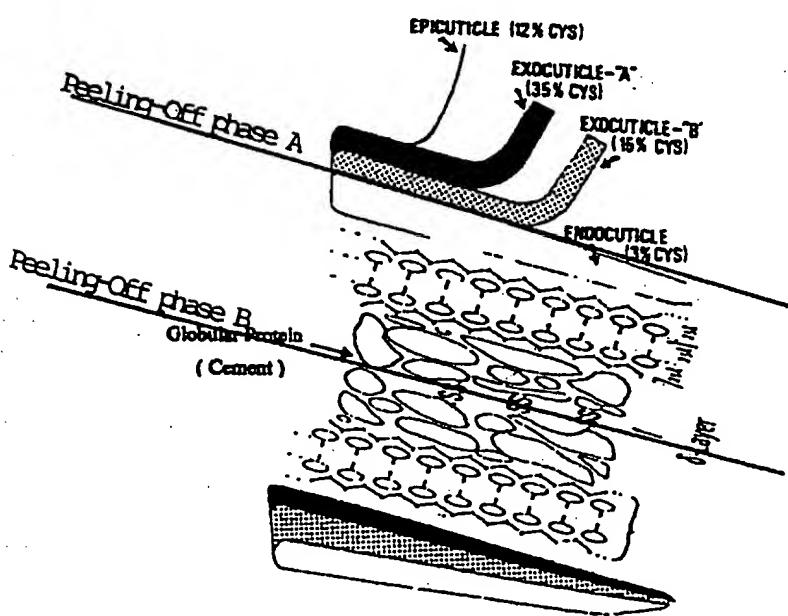
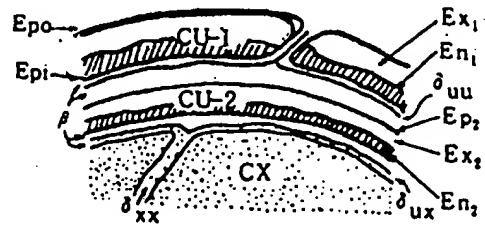


Fig. 1(C) (Y-Y' sectional view)



Cu-1...first cuticle
Cu-2...second cuticle
Ex...Exocuticle
En...Endocuticle
Epo, Epi...outer side Epicuticle
inner side Epicuticle
CU-, cuticle ; **CX-**; cortex
 β , hydrophobic membrane and space
 δ , fillings between cells

Brandbury, Baumann et al. report morphological components in wool proteinaceous chemical components as below.

Table 1-1 Morphological Components in Merino Wool^{a)}

Component	Percent in fiber (mass%)	Cystine crosslink content(mol%) ^{b)}	Remarks
Cuticle			
Epicuticle	0.1	6.0	Keratinous
Exocuticle	6.4	10.0	Keratinous
Endocuticle	3.6	1.6	Non-keratinous
Cortex			
Macrofibrils	74.0	7	Keratinous
Nuclear remnants + intermacrofibrillar material	12.6	1.6	Non-keratinous
Cell membrane complex			
Lipid	0.8		Nonproteinaceous
Soluble protein	1.0	1.1	Non-keratinous
Resistant membrane	1.5	5.0	Keratinous

a) From Bradbury^{3,4)} and Baumann⁶⁾

b) Calculated as cystine + (cysteic acid)/2⁶⁾

J.H.Bradbury, Advan. Protein Chem., 27, 111(1973)

H.Baumann, Fibrous Proteins, Vol.1 (D.A.D.Parry, L.K.Creamer Eds.)
Acadwmic Press, New York p299(1980)

As understood from Table 1-1, Epicuticle and Exocuticle are keratinous. Endocuticle is non-keratinous.

3. WOOL OF HOJO

Hojo et al. peels off keratin layers (epicuticle layer and exocuticle layer) in scales and the endocuticle layer remains, because Hojo et al. describes "The method of this invention enables removal of keratin layers from the scales (outer surface cells)" on lines 10-12 in column 1, "to cause a rapid reaction at the under-keratin layers so that the keratin layers may be removed from the under-keratin layers and the non-keratin protein layers may be exposed" on lines 22 to 26 in column 3, "The method of modifying keratin fiber represented by wool according to the present invention enables peeling off the keratin parts which form scales without damaging the non-keratin protein of the wool" on lines 59-62 in column 5, "Wool fiber was dispersed in the solution and it was appreciated that the water-repellent keratin parts fell off from the fiber" in Examples 1 on lines 31 to 32 in column 6, "it was appreciated that the surface of the fabric became hydrophilic" in Example 2 on lines 30 to 31 in column 7, "the wool fiber became hydrophilic and dispersed in the solution. This shows that the water repellent keratin scales of cuticular cells

"peeled off" in Example 3 on lines 5 to 7 in column 8, "Complete removal of the keratin in cuticles was ascertained by microscopic observation of the fiber" in Example 4 on lines 41 to 42 in column 8, "keratin of the cuticles completely fell off" in Example 5 on line 2 in column 9, "This showed falling off of the water repellent keratin parts" in Example 6 on lines 34 to 35 in column 9, and "Bubbles occurred actively and the bath became turbid and turned brown, which seemed to prove peeling off the keratin layer" in Example 7 on lines 10 to 12 in column 10.

As clearly stated in Hojo et al., keratin layers (epicuticle layer and exocuticle layer) in scales are peeled off and the endocuticle layer remains. The removal of the under keratin layer is not optional as stated by the rejection but rather is the purpose of Hojo's invention. Otherwise, Hojo cannot achieve the aims and effects of his invention. Hojo's invention has effects of not only shrink-proofing but also antibacterial activity.

As described in column 1, lines 15 to 21, "Felting, which is a drawback of wool, results from the deformation caused by the difference in swelling extent due to the difference in water absorbability of keratin layers and non-keratin protein layers constructing the cuticular cells which make-up to the keratin fibers. Various improved methods have been tried to remove the surface cells." The Hojo invention belongs to the field in which the shrink-proofing effect is achieved by removing keratin layers.

As Hojo also discloses, "wool will obtain various useful properties such as antibacterial activity by being subjected to the treatment comprising removing keratin or exocuticles which form the outermost sides of the surface cells of wool fiber to expose endocuticles which are of hydrophilic non-keratin protein" on lines 45 to 50 in column 1. This antibacterial activity can not be obtained unless the hydrophilic endocuticle layers are exposed by removal of keratin layers.

Hojo et al. discloses the invention corresponding to the cited Hojo Patent (U.S. 5,824,113) in:

- 1) H. Hojo and T. Ogura, Proc. 9th Inter. Wool Text. Res. Conf., Biella, Vol. 1, 217 (1995),
- 2) H. Hojo and M. Kamada, Proc. 8th Inter. Wool Text. Res. Conf. Christchurch, Vol. IV, 390 (1990).

The references above are attached herewith.

As can be seen, Hojo refers in reference 1), for example, to "destroy the structure of parts under the exo-cuticle (ex-CU) layer in cuticular (CU) cells" on lines 2 to 3 in SYNOPSIS and

"EX-CU removed wool in Fig. I to Fig. IV. Hojo also refers in reference 2), for example, to "Effects of Removing Exocuticle" in Table III, "EX removed wool" in Fig. 3 and Fig. 4.

4. WOOL OF THE PRESENT INVENTION

In the present invention, the keratin layers (epicuticle layers and exocuticle layers) are not removed. (See the preamble of claims 8 and 9). The animal fiber of the present invention, which is superior in shrink proofing and pilling resistance, is mainly realized by a chemical modification of the epidermal tissue, and the swelling properties of the exocuticle B-layer and the endocuticle layer are made virtually equal to each other with the water repellence property of the uppermost surface being maintained so that, even when dipped into water, the rising of the scales is virtually eliminated. In other words, while the integral structural body of the epicuticle layer and the exocuticle A-layer which is a hard structure in terms of higher density of cystine cross linkage is maintained, that is, while water repellency is maintained, only the exocuticle B-layer is selectively attacked so that its cystine bond, that is, its cross-linking structure, is broken. Only the portions of the surface of the fiber, especially those related to swelling and shrinkage, are subjected to the modification as specified in claims, with the inside of the fiber being protected; therefore, the resulting feature is that the water repellence property of the entire fiber is maintained and the fiber strength is also maintained and endocuticle layers are not exposed (on line 2 to 12 on page 6, on line 9 on page 15 to line 2 on page 16, lines 3 to 22 on page 20, line 25 on page 31 to line 9 on page 32 and line 22 on page 35 to line 5 on page 36 of the present specification).

The animal fiber of the present invention has scales in their original form on the surface of the fiber (see claims 8 and 9) and is oxidized to a degree of oxidation of an -S-S- bond in an epidermal cell of the animal fiber of not less than 0.1 of relative absorbance in an absorption band of -SO₃H group and/or not less than 0.08 of relative absorbance in an absorption band of -S-SO₃Na group (Bunte salts) with an absorbance of an absorption band corresponding to amide I set to 1 in a reflection FT-IR measuring method with the water shrink proofing property and water repellent property that the animal fiber originally possesses.

Hojo's invention cannot achieve water repellence property because the keratin layers are removed and the surface of the fibers are hydrophilic as above described.

The water repellent property affects the hygroscopicity and moisture releasing property and for controlling heat transfer accompanied by adsorption and desorption of water, and exerts

an influence on heat retaining property and comfortability. In other words, the conventional shrink proofing product (including Hojo's invention) can prevent shrinkage (but not very well from the practical viewpoint) by aqueous washing, but lacks heat retaining property and comfortability.

The scale structure with epicuticle layer, exocuticle layer and endocuticle layer incorporated and not destroyed is shown in the present specification as below:

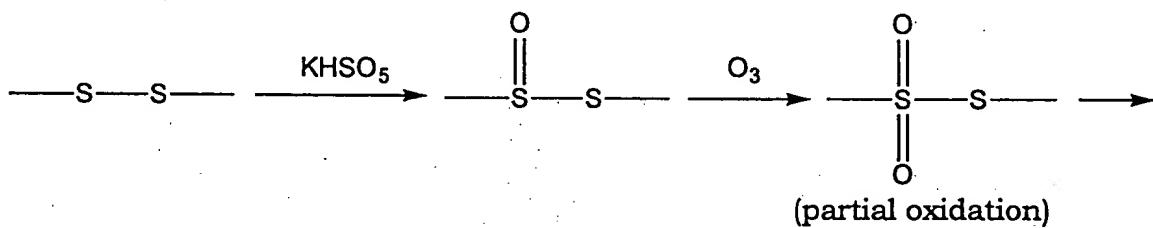
1) evaluation of water repellency by sink-float method to show existence of epicuticle layers (on lines 19 to 25 on page 12 and Table 1 on page 46 in the present specification).

2) Allwörden reaction test to show existence of epicuticle layers (on line 26 on page 12 to line 4 on page 13, lines 10 to 17 on page 49, Fig. 6).

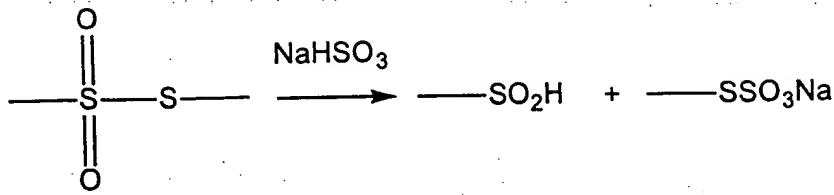
3) photographs of a Scanning Electronic Microscope in Fig. 2 show the same fiber.

In the present invention, the density of the cystine cross-linked bond (-S-S-) in cystine is reduced by an oxidation process and reduction process as shown below, and the difference in water-swelling rate between the exocuticle layer and endocuticle layer is made small, so that arising of scale edge in water can be eliminated.

The present invention pre-oxidizes animal fibers with, for example, potassium hydrogen monopersulfate (PMS), cystine cross-linked bond (-S-S-) is stopped at substantially the mono-oxidized state, and is further oxidized to a higher order using ozone bubbles in the subsequent step (line 4, page 21 to line 17, page 31).



At this oxidation stage, the cystine cross-linked bond (-S-S-) is not cleaved but just partially oxidized. In this partially oxidized state, a shrink proofing effect cannot be obtained. A shrink proofing effect cannot be obtained until a reduction treatment is made to cleave cystine cross-linked bond (-S-S-) (on line 18, page 31 to line 20, page 32).



Unless animal fiber is treated to this stage, the shrink proofing effect cannot be attained in addition to water repellency. This fact is shown in Example 1 and comparative Examples 1 and 2 in Table 1, page 46 in the present specification. Mere oxidation by PMS or ozone bubbles cannot achieve the shrink proofing properties of the present invention even if the proteinaceous fibers are subjected to reduction reaction.

The animal fiber thus obtained can be identified by FT-IR as specified in the present invention as "a degree of oxidation of an -S-S- bond in an epidermal cell of the animal fiber of not less than 0.1 of relative absorbance in an absorption band of -SO₃H group and/or not less than 0.08 of relative absorbance in an absorption band of -S-SO₃Na group (Bunte salts) with an absorbance of an absorption band corresponding to amide I set to 1 in a reflection FT-IR measuring method" (on page 16, line 3 to 19).

5. PRODUCTION METHOD OF HOJO COMPARED WITH THE PRESENT INVENTION

The method of Hojo for modifying keratin fibers is as summarized as below:

- mechanical action is applied to keratin in the presence of aqueous solution of a transition metal salt to weaken and destroy the structure of the part under keratin layers in scales;
- transition metal is introduced in the under keratin layers locally at high concentration;
- then immersed in a bath containing oxidizing agents, i.e. hydrogen peroxide or monopersulfate (PMS), which is contacted with transition metal as a catalyst under alkaline conditions to generate oxygen gas;
- mainly due to pressure of oxygen gas evolved, exocuticle layers are peeled off (for example, on lines 1 to 27 in column 3, and lines 46 to 47 in column 5).

As a result, the keratin layers on scales are destroyed and hydrophilic endocuticle layers are exposed on the surface of treated wool.

Hojo applies mechanical force to wool fibers to deposit transition metal in non-keratin parts (endocuticle). However, because Cell Membrane Complex (CMC) contains much less -S-S- bonding and is much more water-swelling than endocuticle layers, transition metal is liable to deposit in CMC. Therefore, it may be possible that peeling-off occurs at two portions, that is, Peeling-off phase A and Peeling-off phase B as shown in Fig. 1(B) above. In this point, the Examiner's recognition of "the OPTIONAL removal of the under keratin layer" on line 14 on page 4 in the Office Action Summary is correct.

In Hojo, after oxidation treatment, the oxidation-treated wool sliver was immersed in an aqueous solution of 5g/L sodium meta hydrogen sulfite (Examples 1, 3 and 7) and an aqueous solution of 5g/L sodium meta hydrogen sulfite and 10g/L sodium carbonate anhydride (Example 6). As understood as above mentioned, when the oxidation-treated wool sliver was immersed in an aqueous solution of sodium meta hydrogen sulfite, keratin layers have been already peeled off. Hojo uses aqueous solution of sodium meta hydrogen sulfite to inhibit the effect of the oxidizing agent (in Examples 1 to 7).

The rejection states "in Examples 3 and 6, Hojo et al. illustrate the use of a NaCO_3 aqueous solution and the use of NaOH for the purpose of removing the water repellent keratin scales." The purpose of use of Na_2CO_3 or NaOH is to provide an alkaline solution pH of which is adjusted to 10.3 or 9.0, in which oxidizing agents, i.e. hydrogen peroxide or monopersulfate (PMS) can react vigorously with transition metal catalyst at the incorporated region in non-keratin endocuticle layers or CMC in order to generate oxygen gas for peeling off keratin layers. Hydrogen peroxide and monopersulfate (PMS) are unstable in aqueous alkaline conditions and are liable to decompose to generate oxygen gas. Na_2CO_3 or NaOH do not have reduction ability.

In the present invention, PMS is used to oxidize cystine cross-linked bond (-S-S-) to a level of substantially mono-oxidized state. But, this oxidation is carried out under aqueous acidic conditions (pH=2 in the Examples) on lines 17 to 19 on page 26 and on lines 13 to 14 on page 32. Under aqueous acid conditions, PMS etc. are much more stable and less active than under aqueous alkaline conditions.

In the present invention, primary oxidation and ozone oxidation are conducted on the acidic side, because the standard oxidation potential is higher, solubility of ozone in water is higher and the half life is by far longer on the acidic side than the alkaline side.

6. EXPERIMENTAL COMPARISON OF HOJO WITH PRESENT INVENTION

As clearly understood from the above, the product and the production method of Hojo are completely different from that of the present invention.

In addition, attached is Rule 132 Declaration of Kazuhiro NAKASE, one of the present inventors.

In the Declaration, Example 1 of Hojo was traced. The product thus obtained was compared with the product of Example 3 of the present invention, which is representative thereof.

As shown in photograph Fig. 1(C) by a Scanning Electronic Microscope, scales are not observed on the surface of wool fiber treated according to Hojo. The shape which looks like scales in the Hojo's photography is the surface shape of the endocuticle layer. To the contrary, scales are observed as clearly as in the original wool on the surface of wool fiber treated according to the present invention (Fig. 1(A) and Fig. 1(B)).

The existence or non-existence of keratin layer was confirmed by the Allwörden reaction. This result is in conformity with the observation by Scanning Electronic Microscope (see Fig. 3(A)-(C)). Bubbles could be observed in Fig. 3(A) and Fig. 3(B), but not observed in Fig. 3(C).

As shown by the result of water repellency, Hojo's treated wool showed no water repellency (or hydrophilic). To the contrary, the wool treated according to the present invention has the same water repellency as the original wool fiber (see Fig. 2).

7. NONOBVIOUSNESS FROM HOJO AND THORSEN

With respect to Thorsen, it discloses a method for treating proteinaceous animal fiber with aqueous solution of ozone.

The rejection indicates that Thorsen illustrates proteinaceous fibers contacted with a steam-ozone mixture on page 6, lines 5 and 6 of the Official Action.

However, as Example 1 discloses, "An aqueous solution of ozone containing 10.0 mg/1 of ozone was passed into the funnel in the back flow direction at a rate of 31.1 l/min and a

temperature of 27°C", the aqueous solution of ozone was used to oxidize animal fiber, being same in use of aqueous solution of ozone in Examples 2 and 3. Thorsen also disclosed in the SUMMARY OF THE INVENTION that "Of course, the '906 process and the present process also differ in that a stream of steam and an ozone-air (or oxygen) stream are simultaneously impinged upon the fabric in the former method whereas in the latter method the fabric is contacted with an aqueous ozone solution" in column 3, lines 11 to 17.

Therefore, Thorsen's invention is that animal fibers are oxidized in an aqueous solution of ozone.

In Thorsen, fabrics of proteinaceous fibers are contacted with an aqueous solution of ozone at a temperature of about 0-50°C in a concentration of ozone in water of 1-20 mg/l (column 3, lines 21 to 28). Such an oxidation can partly oxidize cystine bonding -S-S- in proteinaceous fibers.

However, Thorsen does not disclose or suggest that an animal fiber as in the present invention simultaneously exhibiting excellent shrink-proofing properties and pilling resistance in addition to water repellent properties can be produced by the claimed combination of primary and secondary oxidation as well as reductive cleavage.

Particularly, as shown with respect to Example 1 and Comparative Example 2 in Table 1, page 46 in the present specification, a mere oxidation by ozone can not achieve the shrink proofing properties of the present invention, even if the proteinaceous fibers are subjected to reduction reaction.

In other words, the excellent shrink proofing properties can not be achieved only by oxidation in such an aqueous solution of ozone as in Thorsen, even if followed by reduction reaction to cleave the oxidized S-S bonding.

Please refer to the felt shrinkage test carried out in the present specification.

It is described in the present specification with respect to the shrinkage test that ① "area shrinkage rate of not more than 8% in a three-hours aqueous washing when measured as a felting shrinkage rate in conformity with Woolmark Test Method 31" (page 6, lines 14 to 18, in the specification), ② "the felting shrinkage rate is measured in conformity with WM TM31 method (Woolmark Test Method 31), and a fabric knitted into a cover-factor C.F. 0.41 with one

thread taken from 14 gages being used as a sample. Here, "conformity to "WM TM31 method" refers to the fact that the measurements were carried out in accordance with the testing procedure of WM TM31 method based upon the ISO 6330 method, while the test washing machine was changed to a Cubex shrinkage testing machine." (page 11, lines 7 to 15).

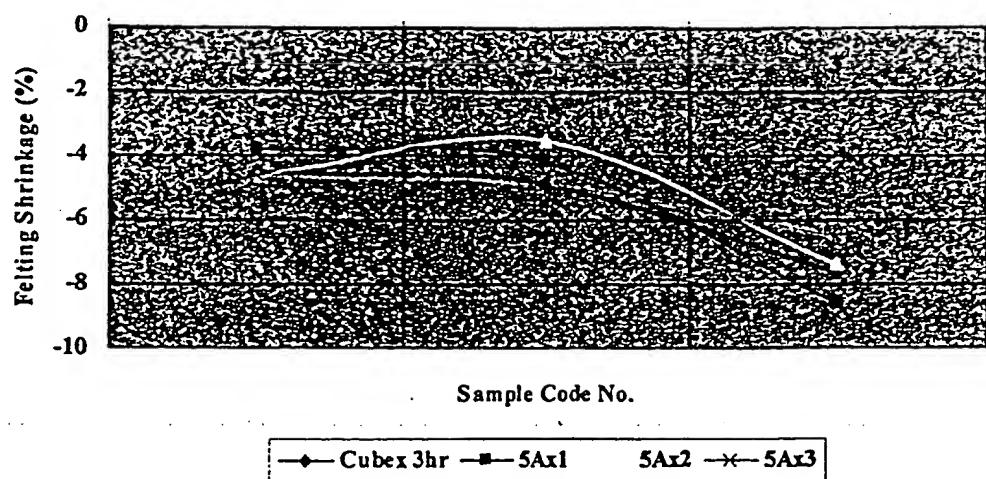
Of record is the manual for the "WM TM31 method". The main modifications of the WM TM 31 test in the present evaluation are in the washing machine, washing times and washing cycles.

In the present invention, a Cubex shrinkage testing machine was used instead of Wascator (in WM TM31), washing time is 3 successive times instead of 12 minutes, and the washing cycle is one.

The modified WM TM31 test in the present evaluation is rather more severe as compared to standard WM TM31 test, as shown below.

By using the Cubex (40x40x40 cm³) International Shrinkage apparatus (specified by IWS TM 185/186 on or about 1981) containing 15L-aqueous washing liquor, the washing time was selected as successive 3 hours at 40°C from Applicants' preliminary test results using the knitted fabric shrink-resisted by the present ozone-treatment, which is indicated in the below Figure. That is, the felting potential by the 15L-Cubex ("Cubex 3hr" line in the figure) is roughly same or rather more severe as compared to ISO 6330 5A x 1 cycle ("5Ax1" line in the figure), ISO 6330 5A x 2 cycle ("5Ax2" line in the figure), or ISO 6330 5A x 3 cycle ("5Ax3" line in the figure) of TM31 using a Wascator shrinkage apparatus.

Relationship between Cubex and Wascator washing methods.



Note: "5A" in the figure means "ISO 6330 5A".

The minus (-) in the figure shows a contraction percentage of the washed knitted fabrics.

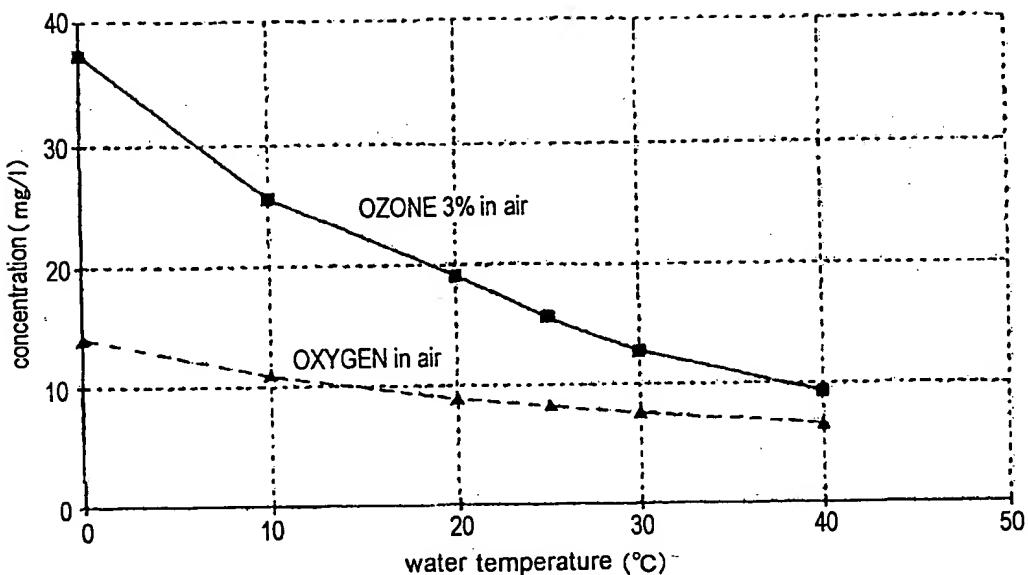
The sample code No. 1, 2, and 3 in the above figure indicates various samples of the knitted fabric composed of the different twist number of 2/48Nm worsted yarn, but the cover factor of the knitted fabrics was 0.41 respectively, i.e., yarn twist of No. 1 was Z480 x S270, No. 2 Z480 x S280 and No. 3 Z480 x S260. (The higher the S value is, the more tightly the fabrics are twisted).

On the other hand, Thorsen carried out shrinkage test in accordance with AATCC Method 124-1973 (column 4, line 50).

This AATCC Method 124-1973 is not practical to evaluate shrinkage properties i.e., from the viewpoint of practical use.

It seems that Official Action recognizes that Thorsen treats proteinaceous fibers with a steam-ozone mixture, but the steam-ozone relates to a conventional technique that Thorsen refers to and has the same low ozone content as an aqueous ozone solution Thorsen uses (lines 51-53 in column 1, lines 15 to 17 in column 3, Examples 1 to 3, etc.).

In Thorsen, fabrics of proteinaceous fibers are contacted with an aqueous solution of ozone at a temperature of about 0-50°C in a concentration of ozone in water of 1-20mg/L (lines 21 to 28, in column 3). Such an oxidation can partly oxidize cystine bonding -S-S- in proteinaceous fibers. In other words, the oxidized degree of proteinaceous fibers is very low, resulting in low shrink proofing property. The low ozone concentration in Thorsen is caused by the low solubility of ozone in itself in water. As shown in Figure below, the solubility of ozone in water is approximately 10mg/L at 40°C.



Even if the steam ozone is used as the rejection states, the steam-ozone mixture has an ozone content of about 10-50 milligrams/liter (mg/L) as low as the ozone oxidation (on lines 51 to 53 in column 1 of Thorsen).

Thorsen describes "advantage of the invention is that the process is carried out at neutral pH, thus, minimizing fiber degradation." This advantage can be achieved using an ozone solution.

To the contrary, Hojo carries out oxidation process under alkaline conditions and keratin layers are removed, being different in those points from Thorsen.

Neither Hojo nor Thorsen discloses reduction treatment to cut the cystine bond (-S-S-).

Even if an ozone solution is applied to the keratin layer removing step, the process is completely different from the present invention. The combined process is just an ozone oxidation process in an ozone solution in the presence of a transition metal catalyst of the under keratin layer.

In addition, as shown with respect to Example 1 and comparative Example 2 in Table 1, page 46 in the present specification, a mere oxidation by ozone bubbles at higher content than Thorsen cannot achieve the shrink proofing properties of the present invention even if the proteinaceous fibers being subject to reduction process.

In other words, the excellent shrink proofing property cannot be achieved only by oxidation at such a low ozone content solution as Thorsen employs, even if followed by reduction reaction to cleave the oxidized S-S bonding.

For the foregoing reasons, it is apparent that the rejection on prior art is untenable and should be withdrawn.

With regard to the rejection of double patenting over claims 7 to 14 of co-pending application Serial No. 09/721,772, the claims of that application and the present application are sufficiently different as to be unobvious in view of each other. In this regard, none of the tests recited in the claims herein are recited in the claims of Serial No. 09/721,772, nor are such claims suggestive of the present claims. Accordingly, the rejection on double patenting is untenable.

No further issues remaining, allowance of this application is respectfully requested.

If the Examiner has any comments or proposals for expediting prosecution, please contact the undersigned at the telephone number below.

Respectfully submitted,

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